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16. ABSTRACT

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JOURNAL ARTICLE

Elements in Major Raw Agricultural Crops in the United States. 2. Other Elements in Lettuce, Peanuts, Potatoes, Soybeans, Sweet Corn, and Wheat

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Six raw agricultural crops (lettuce, peanuts, potatoes, soybeans, sweet corn, and wheat) were collected from fields in major U.S. growing areas and were analyzed for Ca, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Se, and Zn. Statistical frequency distributions of some of the major elements were normal.

In part 1 (Wolnik et al., 1983), the Pb and Cd content of six major agricultural crops was reported based on a large sampling and carefully controlled analytical program. While Cd and Pb are toxic and obviously undesirable in food crops, other major and minor elements are of interest for different reasons. Several elements, such as Ca, Fe, K, Mo, Na, Ni, and P, are essential for human and animal health, and knowledge about their levels in different raw foods will provide information on the nutritional adequacy of diets. Others, such as Cu, Se, and Zn, though essential,

Food and Drug Administration, Cincinnati, Ohio 45202 (K.A.W., F.L.F., R.D.S., and R.W.K.), and Washington, DC 20204 (S.G.C. and G.L.B.), and Soil Conservation Service, U.S. Department of Agriculture, Washington, DC 20013 (M.W.M.).

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Table I. Multielement Recovery and Results for Standard Reference Materials[a]

element	sweet corn amount added, μg/g	sweet corn recovery, %, ± SD[b]	peanuts amount added, μg/g	peanuts recovery, %, ± SD[c]	NBS Spinach 1570 certified, μg/g	NBS Spinach 1570 results, μg/g[d]	NBS Wheat Flour 1567 certified, μg/g	NBS Wheat Flour 1567 results, μg/g[e]
Ca	500	100 ± 2	1000	101 ± 2	13 500 ± 300	12 900 ± 300	190 ± 10	196 ± 3
Cu	10	98 ± 2	10	101 ± 3	12 ± 2	11.8 ± 2	2.0 ± 0.3	2.03 ± 0.05
Fe	10	100 ± 5	10	99 ± 7	550 ± 30	530 ± 11	18.3 ± 1.0	18.6 ± 1.5
K	5000	97 ± 5	5000	103 ± 3	35 600 ± 300	35 200 ± 1000	1360 ± 40	1320 ± 40
Mg	500	97 ± 5	1000	101 ± 4	*f*	8600 ± 200	*f*	401 ± 8
Mn	10	99 ± 2	10	98 ± 4	165 ± 6	165 ± 3	8.5 ± 0.5	8.42 ± 0.23
Mo	10	99 ± 2	10	96 ± 3	*f*	<0.25	0.4[g]	0.396 ± 0.05
Na	400	96 ± 2	*f*	*f*	*f*	*f*	8.0 ± 1.5	*f*
Ni	10	99 ± 2	10	100 ± 2	6[g]	5.51 ± 0.32	0.18[g]	*f*
P	1000	100 ± 4	2000	103 ± 3	5500 ± 200	5300 ± 70	*f*	1390 ± 30
Se	0.05	99 ± 16	0.05	99 ± 16	*f*	*f*	1.1 ± 0.2	0.90 ± 0.05
Zn	10	101 ± 4	10	102 ± 7	50 ± 2	49.8 ± 1.3	10.6 ± 1.0	11.0 ± 0.3

[a] Based on dry weight. [b] Mean of 29–30 recovery determinations. [c] Mean of 21–30 recovery determinations. [d] Mean of 14 determinations. [e] Mean of 29 determinations, except for Se, where mean of 50 determinations. [f] Not determined. [g] Noncertified value.

Table II. Elements in Lettuce[a]

element	μg/g wet weight mean	median	minimum	maximum	CV	normality[b]
Ca	210	170	110	610	48.1	<0.01 S
Cu	0.26	0.24	0.065	0.76	47.2	<0.01 S?
Fe	3.0	2.2	1.3	30	100.7	<0.01 S
K	1700	1500	900	4500	37.7	<0.01 S?
Mg	83	71.5	42.0	210	39.8	<0.01 S?
Mn	1.8	1.25	0.23	11	99.8	<0.01 S
Mo[c]	0.013	0.0067	<0.0039	0.19	202.3	<0.01 S
Na[d]	74	66	14	410	83.2	<0.01
P	220	215	130	490	23.7	<0.01 N1
Se[e]	0.0016	0.00066	<0.0004	0.011	134.4	<0.01 S
Zn	1.9	1.8	0.51	5.1	43.8	<0.01 N1
wwf[f]	0.0415	0.040	0.026	0.084	19.0	

[a] $n = 150$. [b] P value for the Kolomogorov-Smirnov D statistic and frequency distribution as judged by visual observation of the normal probability plot (S = skewed; S? = questionable skewed; N1 = normal except for ≤1% of the data). [c] Sixty-three results below the detection limit. [d] Thirty-four results below the quantitation limit. [e] Fifty-three results below the quantitation limit. [f] Wet weight factor.

have a limited range between required and toxic levels. Other concerns are the interactions between elements, which have been postulated as affecting the toxicity, the level of absorption, and possibly the retention of Cd and Pb by plants (Welch, 1978) and animals (Fox, 1974).

The Food and Drug Administration (Compliance Program Evaluation, 1975, 1978) has determined levels of As, Cd, Hg, Pb, Se, and Zn in a variety of foods. A recent paper from the U.S. Geological Survey (Erdman and Moul, 1982) reported on the analysis of several cultivars of small grains for many elements. An earlier report from the same organization (Shacklette, 1980) presented extensive data on many elements in vegetables, fruits, and soils collected in 1972–1973. In a few other studies, crops (usually a small number of samples) were analyzed for selected elements (Schlettwein-Gsell and Mommsen-Straub, 1970; Lorenz and Loewe, 1977; Thomas et al., 1974; Pfeilsticker and Maskard, 1975; Reith et al., 1974). In none of the studies reported, however, was an attempt made to systematically exclude the effect of human activities to obtain "background" values for these elements. Most work included a limited number of samples of each crop variety so that meaningful statistical data could not be developed. The purpose of this work was to obtain accurate background values for major crops and (later) to investigate correlations with soil element levels, crop species, and other factors that may influence mineral uptake.

MATERIALS AND METHODS

Selection of the sampling sites, techniques for the collection of crop samples, and sample preparation were described in part 1 (Wolnik et al., 1983). Experimental details given below are specific for the determination of Se by hydride generation and for other elements by inductively coupled plasma atomic emission spectroscopy (ICPAES).

Selenium Determination. Portions of the sample composite were digested with a mixture of HNO_3, $HClO_4$, H_2SO_4, and NH_4VO_3 catalyst, and Se was determined by hydride generation/condensation–flame atomic absorption spectrometry according to the method of Hahn et al. (1981). A duplicate sample and spiked sample recovery were carried through the method with every set of 10 samples. Typical recoveries are summarized in Table I.

Multielement Determination. An HNO_3, $HClO_4$, and H_2SO_4 digestion was used for the ICPAES determination of 11 elements (Ca, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, and Zn) in composites of lettuce, potatoes, soybeans, sweet corn, and wheat (Wolnik et al., 1981). For multielement analysis of peanuts, HCl pressure dissolution and ICPAES (Kuennen et al., 1982) were used.

For all six crops, a blank, duplicate sample, and spiked sample recovery were run with every 10 samples. When appropriate, a National Bureau of Standards (NBS) Standard Reference Material was analyzed instead of a spiked sample recovery. A summary of these results is

Table III. Elements in Peanuts[a]

element	μg/g wet weight				CV	normality[b]
	mean	median	minimum	maximum		
Ca	400	380	110	890	40.4	<0.01 N
Cu	7.6	7.25	0.80	19	38.8	<0.01 N1
Fe	18	18	7.8	29	22.8	<0.01 N
K	6600	6500	3300	9900	23.3	>0.15 N
Mg	1600	1700	800	2300	19.7	<0.01 N2
Mn	16	16	3.8	52	28.8	<0.01 N1
Mo[c]	0.43	0.25	<0.032	3.95	129.1	<0.01 S
Ni[d]	2.0	1.4	<0.15	14	107.0	<0.01 S
P	3400	3500	1600	4900	20.7	<0.01 N2
Se	0.057	0.036	0.002	0.91	130.8	<0.01 S
Zn	28	28	12	56	25.7	0.025 N1
wwf[e]	0.8795	0.9515	0.428	0.999	16.0	

[a] $n = 320$. [b] P value for the Kolomogorov-Smirnov-D statistic and frequency distribution as judged by visual observation of the normal probability plot (N = normal; S = skewed; N1 = normal except for <1% of the data; N2 = normal with slight negative skewness). [c] Forty results below the detection limit. [d] Seven results below the detection limit. [e] Wet weight factor.

Table IV. Elements in Potatoes[a]

element	μg/g wet weight				CV	normality[b]
	mean	median	minimum	maximum		
Ca	60	55	17	180	44.9	<0.01 N1
Cu	0.96	0.86	0.14	2.7	52.1	<0.01 S2
Fe	3.65	3.9	1.3	6.6	21.4	0.01 N1
K	4100	4100	2500	5500	11.1	<0.01 N
Mg	200	200	100	290	16.2	<0.01 N
Mn	1.4	1.3	0.72	3.7	34.2	<0.01 S
Mo[c]	0.036	0.027	<0.011	0.27	18.3	<0.01 S
P	500	480	270	890	21.0	<0.01 S?
Se[d]	0.0030	0.0011	<0.002	0.055	176.7	<0.01 S
Zn	3.1	3.0	1.1	7.0	33.0	<0.01 N1
wwf[e]	0.193	0.193	0.126	0.259	12.7	

[a] $n = 297$. [b] P value for the Kolomogorov-Smirnov-D statistic and frequency distribution as judged by visual observation of the normal probability plot (N = normal; S = skewed; S? = questionable skewed; N1 = normal except for <1% of the data). [c] Seventy-three results below the detection limit. [d] One hundred fifty-three results below the quantitation limit. [e] Wet weight factor.

Table V. Elements in Soybeans[a]

element	μg/g wet weight				CV	normality[b]
	mean	median	minimum	maximum		
Ca	2000	1900	1000	3600	20.7	<0.01 N1
Cu	12	12	3.5	29	30.4	<0.01 N
Fe	66	65	48	110	14.7	<0.01 S?
K	18000	18000	14000	21000	6.3	<0.01 N2
Mg	2200	2200	1600	2600	8.7	<0.01 N
Mn	27	24	14	90	30.0	<0.01 S?
Mo[c]	2.1	1.1	<0.06	18	125.7	<0.01
Ni	4.8	4.5	0.35	29	70.2	<0.01 S
P	5500	5500	2900	7900	13.6	<0.01 N
Se	0.19	0.075	0.010	2.5	160.3	<0.01 S
Zn	42	41	29	67	15.5	<0.01 N1
wwf[d]	0.996	0.918	0.816	0.953	1.9	

[a] $n = 322$ except for Se, where $n = 316$. [b] P value for the Kolomogorov-Smirnov-D statistic and frequency distribution as judged by visual observation of the normal probability plot (N = normal; S = skewed; S? = questionable skewed; N1 = normal except for <1% of the data; N2 = normal with slight negative skewness). [c] Nine results below the detection limit. [d] Wet weight factor.

presented in Table I. The recovery values listed for sweet corn are representative of those obtained for potatoes and soybeans. NBS Spinach 1570 and Wheat Flour 1567 were used as controls for the analysis of lettuce and wheat samples, respectively.

Data Processing. The data output from the ICPAES computer was logged onto a magnetic tape data cartridge (Capar and Dusold, 1982). This data tape was read into an IBM 370/168 computer by using the interactive computer language APL (Capar and Dusold, 1978).

Using APL programs, the results were checked for errors and formated onto a disk file. The files were then processed by using the procedure UNIVARIATE of the Statistical Analysis System (SAS) Release 79.5 (SAS, 1979, 1981). SAS was accessed by way of batch processing with job submission through APL. The SAS UNIVARIATE procedure was applied to each crop for each element. This procedure produces a number of simple descriptive statistics including quantiles, vertical bar charts, normal probability plots, skewness, and median. A test for nor-

Table VI. Elements in Sweet Corn[a]

element	μg/g wet weight				CV	normality[b]
	mean	median	minimum	maximum		
Ca	22	17	6.4	91	66.6	<0.01 S
Cu	0.45	0.41	0.19	0.92	36.3	<0.01 S?
Fe	4.0	3.8	1.3	7.5	30.0	<0.01 N
K	2900	2800	2100	4800	10.3	<0.01 N1
Mg	270	270	190	440	18.5	<0.01 N1
Mn	1.6	1.5	0.71	5.6	39.8	<0.01 S
Mo[c]	0.048	0.031	<0.009	0.45	130.1	<0.01 S
Ni[d]	0.062	0.039	<0.026	0.35	82.0	<0.01 S
P	830	820	460	1300	15.5	<0.01 N
Se[e]	0.0064	0.0028	<0.002	0.086	164.7	<0.01 S
Zn	5.6	5.3	2.6	11	28.7	<0.01 S?
wwf[f]	0.214	0.2145	0.083	0.468	28.4	

[a] $n = 268$. [b] P value for the Kolomogorov-Smirnov-D statistic and frequency distribution as judged by visual observation of the normal probability plot (N = normal; S = skewed; S? = questionable skewed; N1 = normal except for <1% of the data). [c] Fifty-six results below the detection limit. [d] One hundred thirty-two results below the detection limit. [e] Fifty-eight results below the quantitation limit. [f] Wet weight factor.

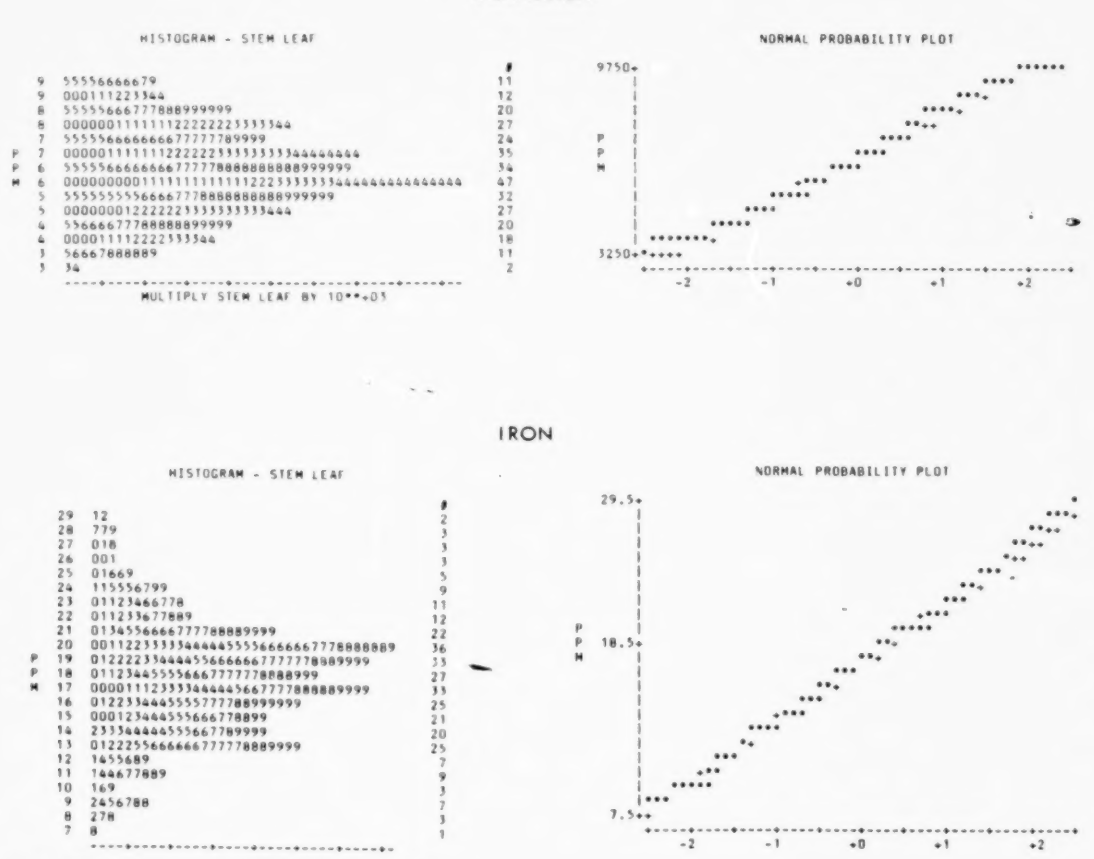

Figure 1. Histogram and probability plot for peanuts.

mality using the Kolomogorov-Smirnov-D statistics (SAS, 1979) was also performed.

The wet weight factor (wwf) reported in the tables can be used to calculate dry weight results (divide values for elements by wwf) or to calculate moisture content (1 − wwf × 100).

RESULTS AND DISCUSSION

Data obtained on 10–11 elements in the six crops are presented in Tables II–VII. Nickel was not determined in lettuce, potatoes, and wheat. Statistical information such as means, medians, and coefficients of variation show differences not only in absolute levels but also in variability. The number of samples for each crop ranged from 150 for lettuce to 332 for soybeans. However, a few elements, especially Mo and Ni, were present at levels below detection limits in some of the crops. For Se, 0.01 μg/g (dry weight basis) was defined as the quantitation level. In the statistical calculations, half of the detection limit value was substituted for sample results that were below the detection or quantitation limit. ICPAES parameters for Na were selected to provide accuracy at high concentration levels. Consequently, except for lettuce, results for Na were below the quantitation limit in the majority of

Table VII. Elements in Wheat[a]

element	μg/g wet weight				CV	normality[b]
	mean	median	minimum	maximum		
Ca	380	370	180	740	23.7	<0.01 N1
Cu	4.4	4.3	2.2	8.7	28.5	<0.01 S
Fe	32.5	32	17	60	22.2	0.07 N1
K	3800	3700	2600	7300	18.5	<0.01 S?
Mg	1400	1400	700	2800	18.3	<0.01 N1
Mn	38	37	13	67	25.5	>0.15 N1
Mo[c]	0.40	0.38	<0.06	1.85	71.2	<0.01 S
P	3400	3400	1400	6000	20.8	0.024 N1
Se[d]	0.37	0.16	<0.010	5.3	163.1	<0.01 S
Zn	27	26.5	9.3	67	35.7	<0.01 N1
wwf[e]	0.881	0.883	0.722	0.988	6.3	

[a] n = 290 except for Cu, where n = 284. [b] P value for the Kolomogorov-Smirnov-D statistic and frequency distribution as judged by visual observation of the normal probability plot (S = skewed; S? = questionable skewed; N1 = normal except for <1% of the data). [c] Twenty-five results below the detection limit. [d] Twenty-three results below the quantitation limit. [e] Wet weight factor.

samples and therefore are not reported.

As shown in Tables II–VII, data for most major elements such as Ca, K, Mg, and P fall within a fairly narrow range, while for trace elements greater differences exist between minimum and maximum values. Although the absolute concentrations of the elements may vary from sample to sample, crop cultivars display distinct patterns of relative elemental abundance even when grown over wide geographical areas. In each of the crops, except lettuce, the general pattern for the major elements is K > P > Mg > Ca. Lettuce follows a K > P > Ca > Mg pattern, although in 33% of the samples, Ca content exceeds that of P. Thirty-five percent of soybean samples display a pattern similar to that of lettuce, in which Ca > Mg, and in 25% of the wheat samples, P > K.

The minor elements display more variation; however, certain predominant patterns are apparent. Sweet corn and peanuts show a Zn > Fe > Mn > Cu pattern except for 30% of the peanut samples, in which Mn > Fe. Only 15 of 320 peanut samples had Cu > Mn. Those 15 samples came from the same state, although the other 49 samples from that state showed the normal pattern.

Soybeans and potatoes give the pattern Fe > Zn > Mn > Cu. Lettuce shows the same general pattern, but in 33% of the samples the pattern changes to Fe > Mn > Zn > Cu, and in 21% of the samples the pattern resembles that in sweet corn and peanuts. The most frequently occurring pattern for wheat is Mn > Fe > Zn > Cu, although 20% of wheat samples show Fe > Mn and 17% show a Mn > Zn > Fe > Cu pattern.

The general trend for trace elements in these crops is Ni > Mo > Se. Nickel was determined only in sweet corn, peanuts, and soybeans, and oftentimes in those crops the Ni > Mo relationship is reversed. In wheat, Se > Mo in 28% of the samples.

The frequency distribution of elements in plant material has been shown to be positively skewed (Shacklette, 1980). This was also true in the present study for some of the elements, especially for trace elements. Some other elements, especially major nutrients such as Ca, K, and P, as well as Fe, Mg, and occasionally Cu and Zn, showed perfect or near perfect linear statistical distributions. This is expressed by the term "normality" in Tables II–VII and is visually presented for Fe and K in peanuts in Figure 1. These graphs were prepared by using an SAS program (SAS, 1981). [Additional examples are provided as supplementary material (see paragraph at end of paper regarding supplementary material)]. As an additional test, plotting some of the data on arithmetic probability paper also resulted in nearly straight lines.

It is not clear what is responsible for the normality of this distribution, which, to our knowledge, has not been reported before. It is possible that the large number of samples collected, prepared, and analyzed by standardized, uniform procedures and the absence of extraneous environmental and other contaminants eliminated the skewing effects observed in other studies. The elements that often showed the most normal distribution were generally those with a relatively narrow range between minimum and maximum values. This limited normal distribution indicates that crop genetic factors are more important than fertility, soil, and environment (Chaney, 1983).

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Registry No. Ca, 7440-70-2; Cu, 7440-50-8; Fe, 7439-89-6; K, 7440-09-7; Mg, 7439-95-4; Mn, 7439-96-5; Mo, 7439-98-7; Na, 7440-23-5; Ni, 7440-02-0; P, 7723-14-0; Se, 7782-49-2; Zn, 7440-66-6.

Supplementary Material Available: Computer printouts containing data tabulations on elements, including Cd and Pb in six crops, and statistical calculations, histograms, and normality plots (119 pages). Ordering information is given on any current masthead page.

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